Journal of Analytical and Applied Pyrolysis 92 (2011) 99–105

Contents lists available at ScienceDirect

Journal of Analytical and Applied Pyrolysis

journal homepage: www.elsevier.com/locate/jaap

Effects of temperature and holding time during torrefaction on the pyrolysis behaviors of woody biomass

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A R T I C L E I N F O

Article history:
Received 1 December 2010
Accepted 21 April 2011
Available online 30 April 2011

Keywords:
Pyrolysis
Torrefaction
Biomass
TG–MS

A B S T R A C T

Torrefaction is the thermal treatment techniques performed at relatively low temperature (<300 °C) in an inert atmosphere, which aims to improve the fuel properties attractively. In this study, woody biomass (*Leucaena leucocephala*) was torrefied at various temperatures and holding times and the pyrolysis behaviors of the torrefied wood were examined in detail by using TG–MS technique. It was found that the carbon content and the calorific value of the torrefied leucaena increased significantly when temperature and holding time during the torrefaction increased. From the TG–MS analysis, the pyrolysis behaviors of the torrefied leucaena were significantly different from those of the raw leucaena. The char yield at 800 °C for the torrefied leucaena was increased when increasing the holding time during the torrefaction. On the other hand, the tar yield during the pyrolysis decreased significantly with the increase in the holding time during the torrefaction. Through the results from the TG–MS analysis, it was concluded that the structure of leucaena was changed by the torrefaction at temperature below 275 °C and the cross-linking reactions occurred during the pyrolysis resulting in increase in char yields and decrease in tar yields. It was also suggested that the longer the holding time during the torrefaction, the more the cross-linking reactions proceed during the pyrolysis. The results obtained from the study provide the basic information for the pyrolyser and/or gasifier design by using torrefied biomass as a fuel.

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1. Introduction

Biomass is a renewable energy resource which is considered as an environmental friendly fuel by producing less CO_2 emission when compared to that of fossil fuel. Especially in Thailand, there are large biomass available in several sectors such as agricultural residue, agro-industries by-products including energy crops for using as alternative energy resources. However, due to its variety of species and compositions which depend on its origin, several technical problems are unavoidably experienced during the biomass utilization. Considering the biomass as feedstock for power generation, particularly in a direct comparison with coal, biomass often reveals undesirable properties. Firstly, in particular, biomass has low energy density, which makes the handling and transportation of biomass very difficult. Secondly, due to the fibrous nature of biomass, it is very difficult to reduce the size of biomass into small particle size especially when biomass is to be used in pulverized system such as co-firing with coal in large scale utility boilers. Moreover, some biomass has high ash content especially alkaline, which will lead to the agglomeration of the bed material inside the reactor as well as the fouling problem found at the surface of water tube in combustion chamber [1–3]. Finally, in most cases, the typical higher moisture content could provide the storage complications such as degradation and also the significant decrease in its heating value. These properties have negative impacts during energy conversion thus resulting in the low gasification/combustion efficiencies and gasifier design limitations.

Several technologies have been proposed to reduce those drawbacks and improve the fuel volumetric energy density. The technologies proposed to address these issues include briquetting/pelletizing which can improve the fuel volumetric energy density [4–7], biochar and bioslurry [8–11], and torrefaction [12–14]. Among these proposed technologies, torrefaction is considered to be very attractive due to its advantage in improved fuel volumetric energy density as well as increased grindability [15,16]. Torrefaction is usually performed in inert atmosphere at temperature below 300 °C which aims to remove mostly the major hemicellulose contents from biomass structure [17,18]. During torrefaction, H_2O and acid compounds were found to be the main condensable volatile products while CO_2 and CO were the major non-condensable gaseous products formation [12]. Prins et al. [13] have studied the weight loss kinetics during the torrefaction. They have showed that the difference in the volatile species released during torrefaction of deciduous wood and coniferous wood was

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doi:10.1016/j.jaap.2011.04.010

Table 1
Structure composition of leucaena used in the study.

Structure composition (wt%, d.a.f.)			
Extractive	Hemicellulose	Cellulose	Lignin
8.0	31.8	33.1	27.1

brought about by the difference in hemicellulose structure. Deciduous wood which mostly contain the acetoxy- and methoxy-group attached to xylose unit in hemicelluloses was suggested to produce more volatile than coniferous wood, especially more methanol and acetic acid.

Moreover, torrefaction is also considered as a pretreatment step for entrained flow gasification because the torrefied biomass, comparing to raw biomass, has high energy density, consumed less energy in size reduction process and the torrefied biomass has more spherical shaped particles during grinding [14,19]. However, few studies have been conducted to examine the effect of torrefaction on the subsequent thermal conversions of torrefied biomass such as pyrolysis, gasification, and combustion.

From previous studies as mentioned, in order to understand and to control the gasification of torrefied wood effectively, the sound understanding of the pyrolysis of the torrefied wood is therefore very necessary. We used *Leucaena leucocephala* as the woody biomass in this study. *L. leucocephala* is a fast growing tree and is considered as a potential energy crop [20]. However, to the authors' best knowledge, the torrefaction of *L. leucocephala* has not been reported so far. So, in this study, the effects of temperature and holding time during the torrefaction on the pyrolysis of *L. leucocephala* have been examined by using TG–MS technique. The results obtained from this study provide the basic information for the pyrolyser and/or gasifier design by using torrefied biomass as a fuel.

2. Materials and experimental methods

2.1. Materials

In this study, woody biomass (*L. Leucocephala*) was selected as a sample. It was firstly shredded with cutting mill and ground with ball mill in order to obtain the sample particle size less than 75 μm. Then, it was dried in vacuo at 70 °C for 24 h before the experiment. The cellulose and lignin analyses in the leucaena are conducted in this study. First, the leucaena sample is extracted for 5–6 h in ethanol in a soxhlet apparatus, according to TAPPI, T264 om-97 [21]. Then, the chemical analyses in the residue after the extraction are conducted. Content of the acid insoluble lignin (known as "Klason lignin") is determined by extracting the residue in a sulfuric acid of 72% (TAPPI T-222) [22]. Content of hemicelluloses and cellulose is determined according to Wise's chlorite method [23] and TAPPI T-203 [24]. The structure composition of leucaena used in this study is shown in Table 1.

2.2. Torrefaction experiments

Fig. 1 shows the schematic diagram of torrefaction experiment. About 30 mg of sample was placed on the quartz wool located at the middle of the reactor (O.D. 10 mm). The nitrogen (99.999% purity) was then purge through the reactor at the flow rate of 40 ml/min. Then the reactor was heated to the desired temperature (200, 225, 250 and 275 °C) at the heating rate of 10 °C/min and held at the desired times. When the desired reaction condition was reached, the gas collected in gas bag was immediately injected to the gas chromatography (Shimadzu, GC-14B) to analyze the gaseous products. After cooling down to room temperature, the solid product or torrefied sample was weighed to measure the yield of torrefied sample.

2.3. Condensables analysis

In order to collect all of condensables as much as possible, the gas bag connected to the end of the reactor was changed to the condenser. The condenser was cooled in the ice bath which was also filled with sodium chloride in order to decrease the temperature to as low as −5 °C. Throughout the torrefaction, condensables attached on the reactor wall and condensed in the condenser was collected carefully by using iso-propanol as a solvent. Prior to analysis, condensables at around 0.5% dilution was initially filtrated by using PTFE membrane filter. About 1 μl of condensables was then injected to GC–MS (Clarus 500, Perkin Elmer) with the helium flow rate of 2 ml/min. The GC column used is Restek1701. On each run, the GC oven was programmed from an initial temperature of 50 °C (5 min) followed by a 10 °C/min increase to final temperature of 250 °C, and held for 5 min. The mass scan spectrum was acquired from 2 to 350 *m*/*z* at a scan rate of 0.1 s.

2.4. Evolved gas analyses during the pyrolysis by TG–MS technique

The pyrolysis experiments were performed in a sensitive thermobalance (Perkin-Elmer, Pyris1 TGA) at a heating rate of 10 °C/min up to a final temperature of 800 °C under the helium flow rate of 50 ml/min. A quadrupole mass spectrometer (Perkin-Elmer, Clarus 500 MS) coupled to the thermobalance (Perkin-Elmer, Pyris1 TGA) was used for the evolved gas analysis. To avoid secondary reactions, a probe was placed very close to the sample pan of the thermobalance in the direction of the gas flow. The transfer lines between the TGA and the MS were heated to 200 °C in order to avoid cold spots and thus prevent the condensation of the gaseous products. The signals for mass numbers of 2, 15, 18, 28, and 44 were continuously detected. Then the mass numbers were converted to the concentrations of H_2, CH_4, H_2O, CO, and CO_2 by referring to the calibration curves constructed using the standard gases. The evolving rates of the gaseous products were estimated from the measurements.

3. Results and discussion

3.1. Changes in weight and gas formation rates during torrefaction

Fig. 2 shows the changes in weight and gas formation rates during the torrefaction of leucaena at 250 °C. The integrated product distributions were also shown in the figure. The amount of tar formed was equated to the difference between the weight loss and the sum of the evolved amounts of H_2O, CO, and CO_2. The weight loss curve is on the dry basis. The weight started to decrease at the temperature above 120 °C and decreased rapidly at around 200–250 °C. After that, the weight decreased gradually when increasing the holding time at 250 °C. The yield of torrefied leucaena at the holding time of 60 min is about 60%. The formation of H_2O, CO, and CO_2 was well accompanied by the rapid decomposition seen in the weight loss curves. It was found that H_2O was the main gaseous product formed at this temperature. It started to evolve at the temperature above 120 °C, had maximum at 250 °C, and then decreased rapidly when increasing the holding time at 250 °C. This H_2O formation at temperature below 250 °C is closely related to the decomposition of hemicellulose as discussed by the previous study [18]. The formation of CO was very little compared with the formation of CO_2. It was found that the yield of condensable volatile products, which hereafter was called

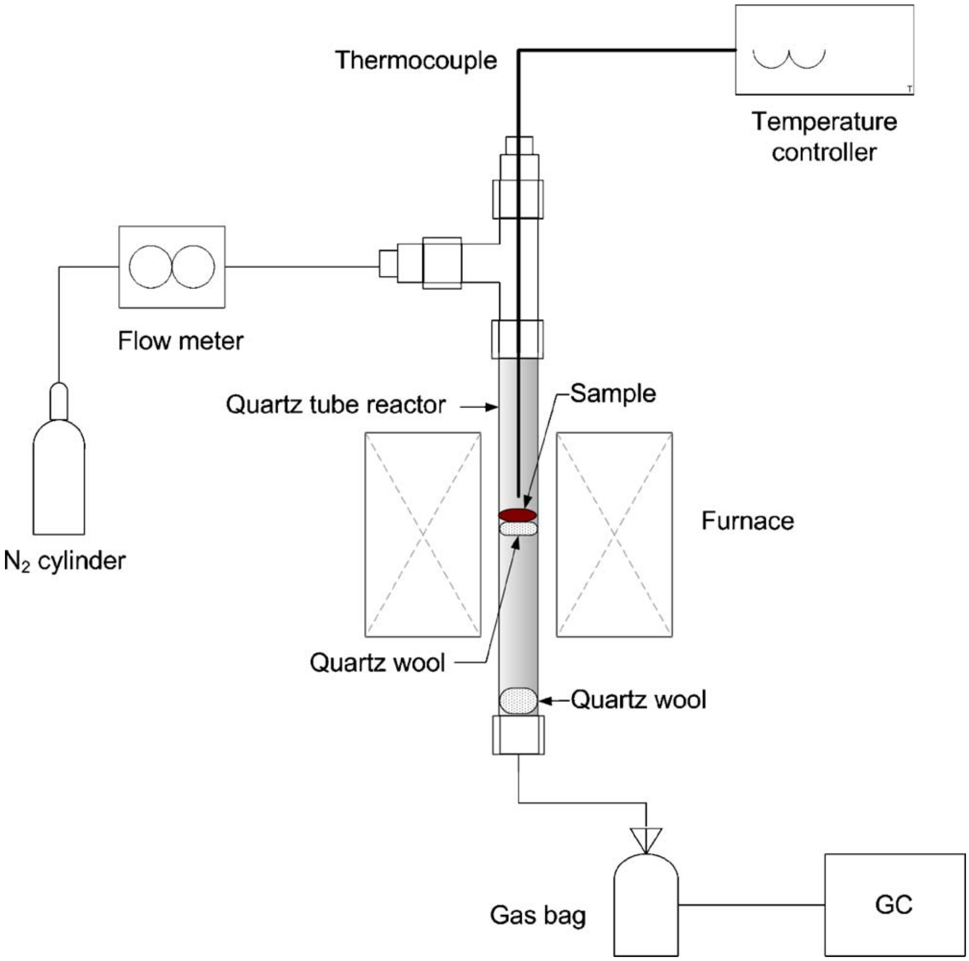

Fig. 1. Schematic diagram of experimental set-up of torrefaction process.

condensables, was as high as 20% during the torrefaction at 250 °C. Fig. 3 shows the GC–MS analysis of these condensables from the torrefaction of leucaena at 250 °C and 275 °C. From the GC–MS analysis, it was confirmed that the main component of the condensables during torrefaction at 250–275 °C was acetic acid. In this study, the increase in torrefaction temperature by 25 °C from 250 °C to 275 °C, the significant increase in the intensity of the condensables components was clearly observed. The effects of temperature and

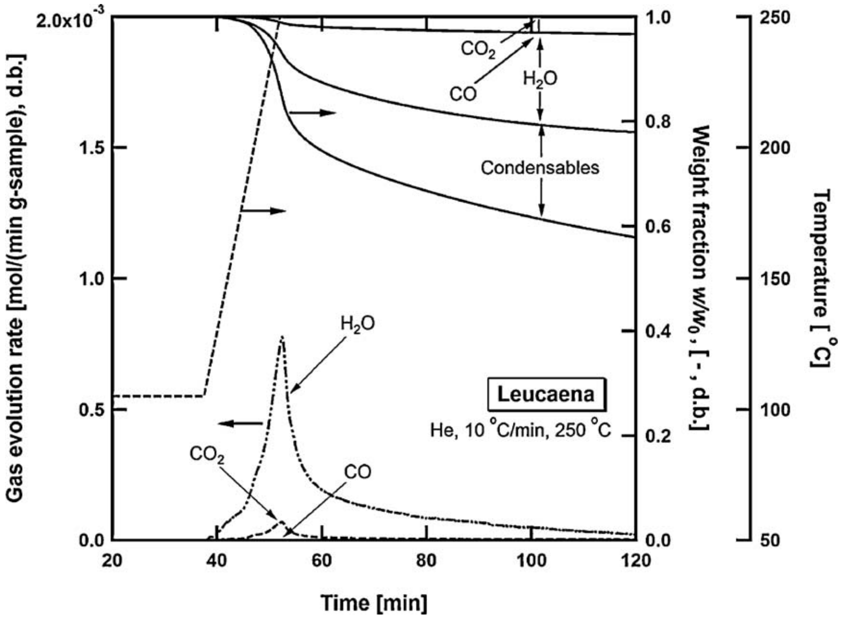

Fig. 2. TG–MS curve during torrefaction at 250 °C of leucaena.

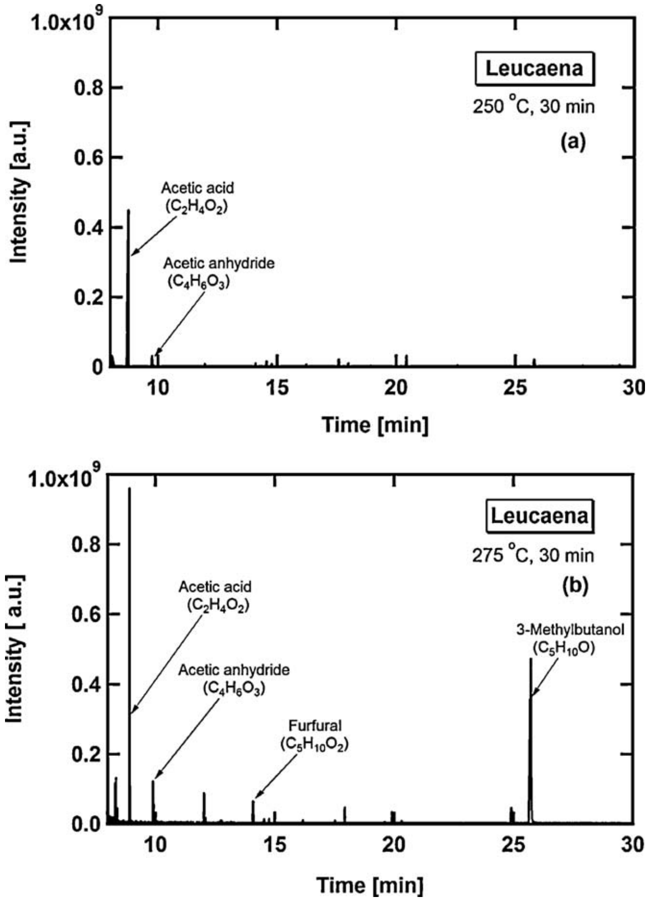

Fig. 3. GC–MS chromatogram of tar produced from torrefaction of leucaena at (a) 250 °C, 30 min-hold and (b) 275 °C, 30 min-hold.

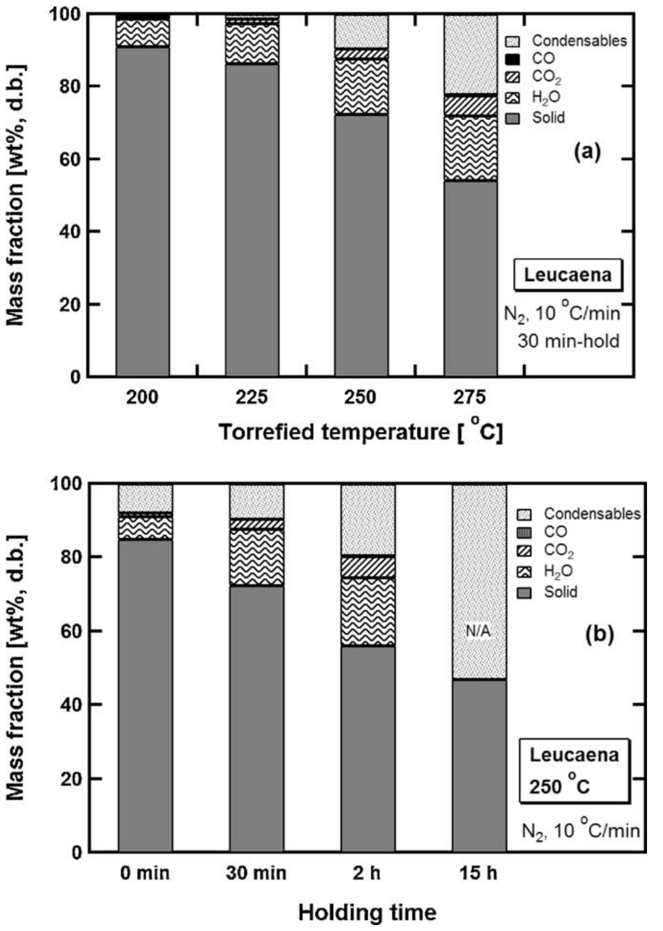

Fig. 4. Product distributions through the torrefaction by varying (a) temperature and (b) holding time.

holding time on the torrefaction will be examined in detail in the next section.

3.2. Yields of products during the torrefaction

Next, we will examine the yields of products (H_2O, CO, CO_2, condensables, and torrefied leucaena) during the torrefaction of leucaena. Fig. 4(a) shows the yields of products during the torrefaction of leucaena at 200–275 °C and holding time of 30 min. The yield of the torrefied leucaena decreased with the increase in the torrefied temperature: it decreased from 91.1% to 54.3% when increases the temperature from 200 °C to 275 °C. It was found that the main gaseous product was H_2O. The yield of H_2O was increased from 7.6% to 17.8% when the temperature increases from 200 °C to 275 °C. The condensables yield at 200 °C was very small: it was only 0.6%. However, the condensables yield increased significantly when increases the temperature. The condensables yield at 275 °C was as high as 22.1%. The yields of CO and CO_2 were very small for the torrefaction at this range of temperature. Fig. 4(b) shows the yields of products during the torrefaction at 250 °C and holding time of 0 min–15 h. It was found that the yield of torrefied leucaena decreased significantly with the increase in the holding time. The yield of torrefied leucaena decreased from 85.0% to 47.0% when increase the holding time from 0 min to 15 h. The condensables yield increased significantly with the increase of holding time: it increased from 7.9% to 19.6% when increases the holding time from 0 min to 2 h. The yield of H_2O increased from 6.1% to 9.5% when the holding time increases from 0 min to 30 min, but it did not increase when the holding time was further increased to 2 h. The yield of CO_2 increased from 1.0% to 6.6% when the holding time increased from 0 min to 2 h. In this study, the collection of gaseous product for the torrefaction at the holding time of 15 h was unavailable due to the limitation of gas bag capacity, however, the increasing tendency of gaseous products and condensables with the increase in the holding time could be predicted.

The proximate analyses, ultimate analyses, and the calorific values of the torrefied leucaena at various conditions are shown in Table 2. It was found that the volatile matter as well as the oxygen content of the torrefied leucaena decreased significantly with the increase in temperature and holding time during torrefaction. On the other hand, the fixed carbon, ash and carbon content of the torrefied leucaena increased with the increase in temperature and holding time during the torrefaction. These results indicated that the oxygen functional groups in the leucaena were decomposed resulting in the higher yield of H_2O and CO_2 with the increase in temperature and holding time during the torrefaction as discussed in the previous section. The carbon content and oxygen content of the leucaena torrefied at 250 °C and holding time of 15 h were 61.3% and 32.3%, respectively, which resulted in the higher heating value of 24.4 MJ/kg. This result indicated that the calorific value of the leucaena could be increased more than 20% when the holding time increased during the torrefaction to 15 h. Table 3 shows the mass energy density of leucaena torrefied at various conditions. The mass energy density is defined as follows:

$$\text{mass energy density} = \frac{\text{energy yield}}{\text{mass yield}}$$

Table 2
Chemical properties and solid yields of leucaena and leucaena torrefied at various conditions.

Sample	Proximate analysis (%, d.b.)			Solid yield (%, d.b.)
	Volatile matter	Fixed carbon	Ash	
Leucaena	86.1	13.1	0.8	100.0
Torrefaction				
200 °C, 30 min	85.3	14.0	0.7	91.0
225 °C, 30 min	84.3	14.9	0.8	86.5
250 °C, 30 min	82.2	16.9	0.9	73.0
275 °C, 30 min	73.8	24.9	1.3	54.5
250 °C, 2 h	76.6	22.1	1.3	56.1
250 °C, 10 h	69.8	28.9	1.4	51.2
250 °C, 15 h	61.8	36.7	1.5	47.0

Sample	Ultimate analysis (%, d.a.f.)				HHV
	C	H	N	O (diff.)	(MJ/kg, d.b.)
Leucaena	50.1	7.4	0.7	41.8	20.3
Torrefaction					
200 °C, 30 min	51.7	7.1	0.7	40.5	21.0
225 °C, 30 min	52.4	7.1	0.7	39.8	21.2
250 °C, 30 min	53.0	6.4	0.7	39.9	21.2
275 °C, 30 min	57.2	5.5	0.8	36.5	22.8
250 °C, 2 h	53.8	5.6	0.8	39.8	21.2
250 °C, 10 h	58.6	5.2	1.0	35.2	23.3
250 °C, 15 h	61.3	5.4	1.0	32.3	24.4

where

$$\text{energy yield} = \frac{\text{mass yield} \times \text{HHV}_{\text{torrefied sample}}}{\text{HHV}_{\text{raw sample}}}$$

It was found that the mass energy density of the torrefied leucaena increased with increasing temperature and holding time. The mass energy density of the torrefied leucaena increased from 1.03 for the torrefaction at 200 °C and 30 min to 1.12 when torrefaction at 275 °C and 30 min. Whereas the mass energy density of the torrefied leucaena increased to 1.20 when the holding time increased to 15 h at 250 °C. By considering the yield of torrefied leucaena and the mass energy density, the torrefaction at 275 °C and 30 min is enough. The values of mass energy density for other biomass samples are also compared in Table 3. These results indicated that the mass energy density of the leucaena could be controlled by carefully adjusting the temperature and holding time during the torrefaction.

3.3. Pyrolysis behaviors of the torrefied leucaena

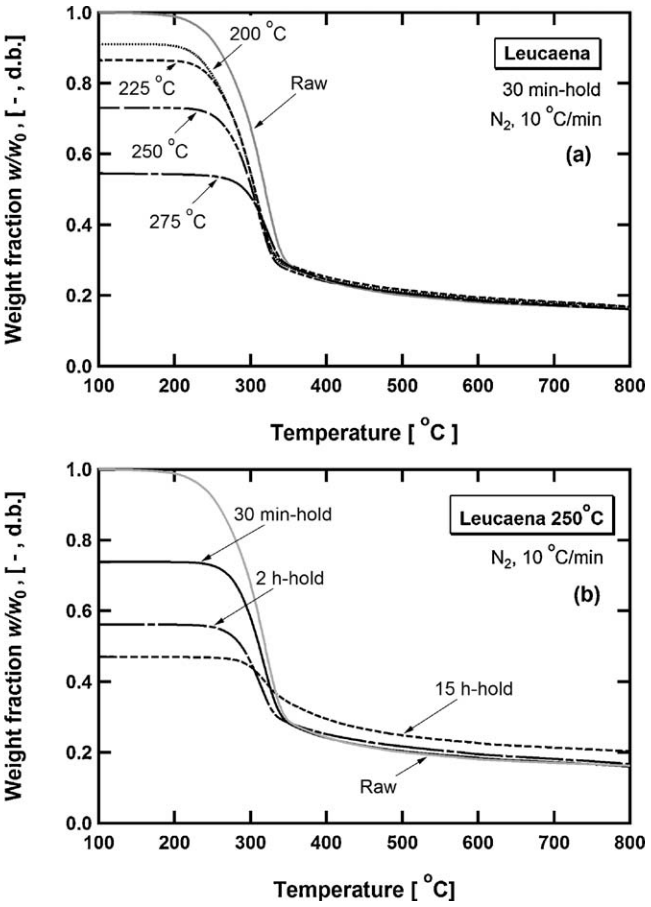

Fig. 5. Weight changes during pyrolysis of raw and torrefied leucaena prepared at various (a) temperatures and (b) holding times.

Fig. 5 shows the weight changes during the pyrolysis of raw and the torrefied leucaena preparing at various torrefaction conditions: at temperatures 200–275 °C (Fig. 5(a)) and at holding times of 30 min to 15 h (Fig. 5(b)). In order to compare with the raw leucaena, the weights of the torrefied leucaena were normalized by multiplying the weights with the values corresponding to the torrefaction yields. From the experiments, the weight change behaviors of the torrefied leucaena were significantly different from the raw leucaena. The weight of raw leucaena started to decrease at above 120 °C, while the weight of the torrefied leucaena started to decrease at temperature higher than the torrefaction temperature. For example, the weight of leucaena torrefied at 275 °C started to decrease at temperature higher than 275 °C. The weight of raw leucaena decreased rapidly at 200–350 °C. After that, the weight decreased gradually when increasing the temperature further from 350 °C to 800 °C. The weight of the torrefied leucaena decreased more gradually than the raw leucaena at 250–350 °C. Moreover, it was found that the char yield at 800 °C for the torrefied leucaena at 200–275 °C and the holding time of 30 min was almost the same as that of the raw leucaena. On the other hand, the char yield at 800 °C for the torrefied leucaena increased with the increase in holding time: the char yield increased from 16.1% to 16.6% and 20.2% when increasing the holding time from 30 min to 2 h and 15 h at torrefaction temperature 250 °C, respectively. These results suggested that the holding time of 30 min during the torrefaction was not enough to increase the char yield of the torrefied leucaena. The increase in the char yield could be explained by the progress of cross-linking reactions during the pyrolysis of the torrefied leucaena. Through these results from the TG analysis, it was concluded that the structure of leucaena was changed by the torrefaction at

Table 3
Mass energy density of torrefied leucaena and other torrefied biomass.

Sample	Torrefied condition	Mass energy density	Reference
Leucaena	200 °C, 30 min	1.03	
	225 °C, 30 min	1.04	
	250 °C, 30 min	1.04	
	250 °C, 2 h	1.04	
	250 °C, 10 h	1.15	
	250 °C, 15 h	1.20	
	275 °C, 30 min	1.12	
Larch	250 °C, 30 min	1.05	[13]
	270 °C, 15 min	1.08	
	250 °C, 30 min	1.11	
Willow	250 °C, 60 min	1.12	[13]
	270 °C, 30 min	1.18	
Beech	240 °C, 30 min	1.12	[13]
	250 °C, 30 min	1.13	
	280 °C, 30 min	1.19	
Straw	250 °C, 30 min	1.15	[13]

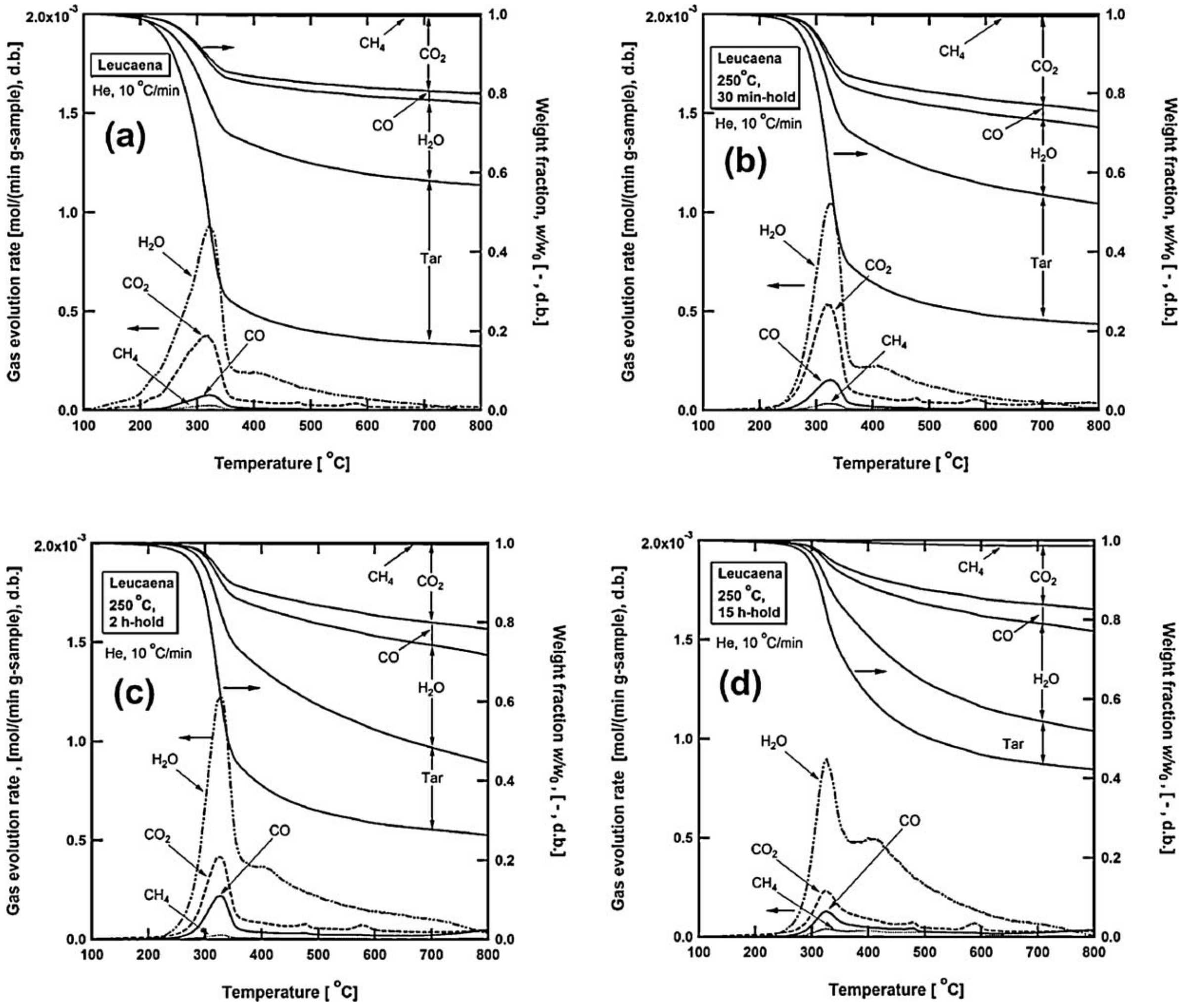

Fig. 6. TG–MS curves of leucaena and torrefied leucaena pyrolysis; (a) leucaena, (b) leucaena torrefied at 250 °C, 30 min-hold, (c) leucaena torrefied at 250 °C, 2 h-hold and (d) leucaena torrefied at 250 °C, 15 h-hold.

temperature below 275 °C and the cross-linking reactions occurred during the pyrolysis resulting in increased in char yields.

3.4. TG–MS analyses of the torrefied leucaena during the pyrolysis

Next, we will examine the pyrolysis behaviors of the torrefied leucaena in detail by TG–MS technique. The changes in weight and gas formation rates during the pyrolysis of raw leucaena and leucaena torrefied at 250 °C and various holding time are shown in Fig. 6. The integrated product distributions were also shown in the figure. It was found that the weight decreasing profiles and the gas formation rates were significantly different among the samples. The major weight decrease was observed at around 250–350 °C for all the samples. The formation behavior of CH_4, H_2O, CO, and CO_2 was well accompanied by the sharp decomposition observed in the weight loss curves. It was found that H_2O was the main gaseous product for all the samples. In particular, H_2O started to evolve above 120 °C for the raw leucaena, while it started to evolve above 200 °C for the torrefied leucaena. The profile of H_2O formation rates had two peaks at 310 and 400 °C for all the samples. The height of the peak at around 310 °C decreased, while the height of the peak at around 400 °C increased with the increase the holding time during the torrefaction. On the other hand, the amount of CO_2 evolved through the pyrolysis decreased with the increase in the holding time during the torrefaction: it decreased from 20.5 wt% for raw leucaena to 16.7 wt% for leucaena torrefied at 250 °C and 15 h. Moreover, it was found that the tar yield during the pyrolysis decreased significantly with the increase in the holding time during the torrefaction. It decreased from 41.9 wt% for raw leucaena to only 7.6 wt% for leucaena torrefied at 250 °C and 15 h. In order to compare with the raw leucaena, the product distribu-

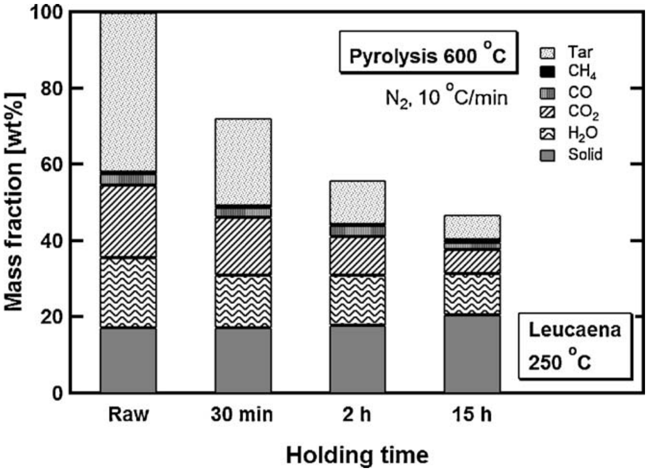

Fig. 7. Product distribution through the pyrolysis at 600 °C of the leucaena torrefied at 250 °C and various holding times.

tion during the pyrolysis at 600 °C of torrefied leucaena at 250 °C and various holding times was re-calculated on the raw leucaena basis and is shown in Fig. 7. It was found that the tar yield and CO_2 yield during the pyrolysis decreased significantly when the holding time increased during the torrefaction. On the other hand, the char yield gradually increased when increase in the holding time during the torrefaction. It was suggested that the structure of leucaena was changed by the torrefaction and it clearly showed that the cross-linking reactions occurred during the pyrolysis of the torrefied leucaena resulting in increase in char yield and decrease in tar yield. It was also suggested that the longer the holding time during the torrefaction, the more the cross-linking reactions proceed during the pyrolysis.

4. Conclusions

Leucaena was torrefied at various temperatures and holding times and then the effect of the torrefaction on the pyrolysis behaviors of the torrefied leucaena was examined in detail by using TG–MS technique. It was found that the carbon content and the calorific value of the torrefied leucaena increased significantly when increase the temperature and holding time during the torrefaction. Condensables was the major product evolved during the torrefaction followed by H_2O, CO_2 and CO, respectively. From the TG–MS analysis, the pyrolysis behaviors of the torrefied leucaena were significantly different from those of the raw leucaena. The char yield at 800 °C for the torrefied leucaena was increased when increasing the holding time during the torrefaction. On the other hand, the tar yield during the pyrolysis decreased significantly with the holding time increased during the torrefaction. Through the results from the TG–MS analysis, it was concluded that the structure of leucaena was changed by the torrefaction at temperature below 275 °C and the cross-linking reactions occurred during the pyrolysis resulting in increase in char yields and decrease in tar yields. It was also suggested that the longer the holding time during the torrefaction, the more the cross-linking reactions proceed during the pyrolysis. The results obtained from the study provide the basic information for the pyrolyser and/or gasifier design by using torrefied biomass as a fuel.

Acknowledgments

The financial support from the Thailand Research Fund and the National Research University Project throughout this study is gratefully acknowledged. The authors would like to acknowledge the PTT public company and Prof. S. Tudsri from Kasetsart University for providing the Leucaena samples used in this study.

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